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Pozzobon, João Luiz ; Missau, Taiane ; Druck, Carolina Ceolin ; Özcan, Mutlu ; Valandro, Luiz Felipe

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Effects of different particle deposition parameters on adhesion of resin cement to zirconium dioxide and phase transformation

João Luiz Pozzobon, DDS, MSciD[a] / Taiane Missau, DDS, MSciD[a] / Carolina Ceolin Druck, DDS, MSciD[b] / Mutlu Özcan, Dr.med.dent., PhD[c] / Luiz Felipe Valandro, DDS, MSci, PhD[d]

[a]MSciD (c), Graduate Programs in Oral Science, Prosthodontics Unit, Faculty of Odontology, Federal University of Santa Maria, Santa Maria, Brazil.

[b]MSciD, Graduate Programs in Oral Science, Prosthodontics Unit, Faculty of Odontology, Federal University of Santa Maria, Santa Maria, Brazil.

[a]Professor, University of Zurich, Dental Materials Unit, Center for Dental and Oral Medicine, Clinic for Fixed and Removable Prosthodontics and Dental Materials Science, Zurich, Switzerland.

[d]Associate Professor, MSciD/PhD Graduate Programs in Oral Science, Prosthodontics Unit, Faculty of Odontology, Federal University of Santa Maria, Santa Maria, Brazil.

Short title: *Bond to zirconia after silica coating parameters*

Correspondance to: *Luiz Felipe Valandro, D.D.S., M.Sci.D., Ph.D., Associate Professor, Federal University of Santa Maria, Faculty of Odontology, Head of Ph.D.-M.Sci.D. Graduate Program in Oral*

Science, Prosthodontics Unit, R. Floriano Peixoto, 1184, 97015-372, Rio Grande do Sul State, Santa Maria, Brazil. Tel: +55-55-3220-9276, Fax: +55-55-3220-9272. e-mail: lfvalandro@hotmail.com

Abstract: This study evaluated the effect of air-abrasion parameters such as particle size, distance and time on adhesion of resin cement to zirconium dioxide (Y-TZP) and t→m phase transformation. Y-TZP blocks (N=80) (In-Ceram YZ, Vita) (4x4x3mm^3) were assigned into eight groups (n=10): air-abrasion with 30μm (CoJet Sand, S30) and 110μm (Rocatec-Plus, S110) silica-coated alumina particles, applied for either for 10 or 20 seconds (T=time), from a distance of 10 or 20 mm (D=distance), composing the following groups: $S_{30}T_{10}D_{10}$, $S_{30}T_{10}D_{20}$, $S_{30}T_{20}D_{10}$, $S_{30}T_{20}D_{20}$, $S_{110}T_{10}D_{10}$, $S_{110}T_{10}D_{20}$, $S_{110}T_{20}D_{10}$, and $S_{110}T_{20}D_{20}$. Resin composite (RelyX ARC) was bonded to Y-TZP blocks in polyethylene moulds. The specimens were aged (10.000 thermal cycles and water storage for 90 days) prior to shear bond test. Failure types were analyzed under stereomicroscope and SEM, and phase transformation was calculated. Data (MPa) were analyzed using 3-way ANOVA and Tukey`s tests. Air-abrasion with 110μm silica particles (10.96) presented significantly higher bond strength (p=0.0149) compared to 30μm (8.96). Time (p=0.403) and distance (p=0.179) parameters did not affect the results significantly. Air-abrasion with 110μm particles (12.3) promoted higher bond strength than that of 30μm (6.4) when applied for 10s from a distance of 10mm (Tukey`s). Failure types were predominantly adhesive. Phase transformation ranged between 30.3 and 35.9% for 30μm particles, and 23.8 to 43.7% for 110μm particles. While the size of silica-coated alumina particles were more relevant parameter for resin cement adhesion to Y-TZP, time (up to 20s) and distance (up to 20mm) appear be less pertinent.

Keywords: Adhesion, air-abrasion, phase transformation, zirconium dioxide

Introduction

Due to its superior mechanical properties such as high flexural strength, fracture toughness, its biocompatibility and favorable optical properties, Yttria-stabilized tetragonal zirconia (Y-TZP) is currently a material of great interest in dentistry [1]. With the introduction of CAD/CAM technology, Y-TZP was used as orthodontic brackets, endodontic posts [1,2] for frameworks of fixed dental prosthesis (FDP) and for monolithic restorations [3]. Y-TZP presents a high crystalline content, consisting of 0.2-0.5 µm diameter grains that are prepared from fine ZrO_2 particles and 3.5-8.7% yttrium oxide Y_2O_3 [4]. Y-TZP is available in three crystalline forms: monoclinic, tetragonal and cubic. At room temperature, pure zirconium dioxide is found in the monoclinic phase that is stable in this phase up to 1170°C. Above this temperature, it transforms into tetragonal and cubic phases [5]. The tetragonal to monoclinic phase transformation is followed by volume increase of up to 3-5%, causing high internal stresses. In order to control this volumetric alteration and stabilize Y-TZP in its tetragonal phase at the room temperature, Y_2O_3 is added [1]. When this material is exposed to stresses during milling for instance, compressive stresses occur around the crack tip. However, transformation from tetragonal to monoclinic phase prevents such crack propagation accompanied by increase in fracture toughness. Yet, this transformation mechanism does not eliminate the crack progression completely [5,6].

In spite of its excellent mechanical properties, Y-TZP does not exhibit high adhesive potential. Considering its microstructure and chemical composition, this ceramic is classified as acid resistant or non-etchable ceramic, meaning that hydrofluoric acid cannot modify its surface, due to lack of glassy phase in its composition [7]. In acid-sensitive or etchable ceramics on the other hand, hydrofluoric acid etching selectively dissolves the glassy phase, enabling micromechanical

retention [8]. Hence, in order to improve adhesion of resin cements to Y-TZP, alternative surface conditioning methods are suggested. The most commonly accepted surface conditioning method is the use of air-abrasion protocol with silica coated aluminum oxide particles that creates a rough surface and increase the mechanical retention [7,8]. This conditioning step is followed by the application of a silane coupling agent or primer based on methacryloxyprophyl trimetoxysilane (MPS) [9]. Such physicochemical activation of the surface is called tribochemical silica coating. Two types of silica particles are frequently used in dentistry, one designed for laboratory (110 μm Rocatec-Plus, 3M ESPE) and the other for chairside (30 μm CoJet, 3M ESPE) use [10]. The purpose of both air-abrasion options is to promote silicon oxide coating on the ceramic surface, making the surface chemically more reactive to silane coupling agents [7], increasing the surface roughness and area for adhesion [11], modifying the surface energy, and consequently increase the resin bond to Y-TZP surface [12].

Application of silane coupling agent is essential for chemical bonding to Y-TZP. Silane molecules react with water forming silanol groups (–Si–OH) from corresponding methoxy groups (–Si–O–CH_3). Siloxane bonds (–Si–O–Si–O–) are then formed between silanol molecules and silica coated surface on one side and on the other side with methacrylate groups of the resin cements [7,13].

Even though tribochemical silica coating increases the chemical and micromechanical adhesion [7,8,11], some studies have shown that air-abrasion could introduce microcracks in the Y-TZP, decreasing its strength [14,15]. On the contrary, other studies have reported an increase in mechanical strength of such ceramics after air-abrasion [16,17]. The controversy between these studies could be explained by the distinct variation in parameters employed during air-abrasion, such as particle type and size, pressure and duration of application, distance and angle of the nozzle to the surface [2,6-8,17-19]. Coating Y-TZP ceramic with 110 μm silica particles were found to be more effective than with 30 μm [19] which at the same time yield to increased phase

transformation [19]. Özcan et al. reported that the angle of the nozzle to the substrate surface during air-abrasion also significantly influences the quantity of silica deposited on the surface of Y-TZP [18]. However, the nozzle distance between the air-abrasion device and the substrate seemed to have less effect [18]. Since silica coating is a useful surface conditioning method to increase adhesion to Y-TZP [20], it is relevant to propose effective deposition parameters that could increase adhesion without increasing monoclinical phase of Y-TZP ceramics.

The objectives of this study therefore, were to investigate a) the effect of air-abrasion parameters including particle size, distance and time of deposition, on the adhesion of resin cement to Y-TZP zirconia and b) their impact on phase transformation. The null hypothesis tested was that deposition parameters used for air-abrasion would not influence the adhesion to Y-TZP and phase transformation.

Materials and Methods

Specimen production

Y-TZP ceramic blocks (VITA In-Ceram 2000 YZ cubes for inLab, Vita Zahnfabrik, Bad Säckingen, Germany) were obtained using a cutting machine (IsoMet 1000, Buehler, Lake Bluff, USA). The resulting pre-sintered blocks (N=80, n=10 per group) had adhesive surface area of 6 mm x 6 mm x 5 mm. The ceramic surfaces were finished using Sof-Lex disks (3M ESPE, Seefeld, Germany) and ground with 1200-grit silicone carbide paper under water-cooling (3M ESPE, Seefeld, Germany). They were then sintered in a programmed furnace according to the manufacturer`s instructions. Y-TZP specimens (4 mm x 4 mm x 3 mm) were then embedded in plastic moulds (h=14 mm, Ø=25 mm) using acrylic resin (Jet-Clássico, Artigos Odontológicos Clássico, São Paulo, Brasil), keeping the upper surface free for bonding.

The specimens were randomly assigned into eight groups (n=10), considering the air-abrasion parameters, namely particle size in 2 levels (30 and 110 μm), time in 2 levels (10 and 20 s), and distance in 2 levels (10 and 20 mm) (Table 1).

Composite cylinder production

Resin composite cylinders (Opallis, FGM, Joinville, Brazil) were fabricated for each ceramic block, using a split metal mould (Ø=3.25 mm; h=3 mm) in two increments. Each layer was photo-polymerized for 40 s (Radii-cal, SDI, Bayswater, Australia; light output: 1200 mw/cm^2), and then further polymerized for 20 s from four directions after removal of the mould. The bonded adhesive area was 8.3 mm^2.

Surface conditioning methods and bonding procedures

The specimens were ultrasonically cleaned (Vitasonic, Quantrex 90, L&R Ultrasonics, Kearny, NJ, USA) in isopropyl alcohol for 10 minutes prior to air-abrasion protocols. Particle deposition was achieved perpendicular to the surface at a pressure of 2.8 bar using a custom made device to standardize the application. After air-abrasion, MPS silane coupling agent (ESPE-Sil, 3M ESPE, St. Paul, USA) was applied to the specimen surfaces using a clean microbrush each time, and allowed to react for 5 minutes.

Resin cement (RelyX ARC, 3M ESPE) was mixed according to the manufacturer`s recommendation, and applied in the cylinders on the specimen surface. Excess cement was removed with a brush, photo-polymerized (Radii-cal) for 20 s from the top and for 40 s from four directions at the bonded area.

Aging process and shear bond test

The specimens were initially subjected to thermal cycling (x10.000 cycles, 5-55°C, dwelling time: 30 s) (Ethic Technology, Vargem Grande Paulista, Brazil) and then stored in distilled water at 37°C for 90 days.

The bonded specimens were loaded under shear in a Universal Testing Machine (EMIC, São José dos Pinhais, Brazil) using a wire loop (Ø=0.12 mm) at a cross-head speed of 1 mm/min. The bonded interface was loaded until failure and the bond strength ***R*** (MPa) was calculated according the formula R=F/A, where ***F*** is the load for debond (N), and ***A*** is the cross-sectional area (mm^2).

Failure type analysis

All debonded specimens were observed under stereomicroscope at x15 (Discovery V20, Carl Zeiss, Gottingen, Germany) to analyze the failure pattern. Failure types were classified as follows: *Adhesive*: Failure at the ceramic-cement interface predominantly; *Cohesive*: Cohesive failure in the resin cement. Representative failures were selected and analyzed under Scanning Electron Microscopy (SEM, JSM-6360, JEOL, Tokyo, Japan).

Micro-morphological analysis

Two additional Y-TZP specimens from each surface conditioning protocol were evaluated under SEM (x1000 to x10000) in order to observe the topographical changes after air-abrasion.

X-Ray diffraction analysis

Quantitative analysis of phase transformation was calculated (n=2 per group) in order to determine the relative amount of m-phase and depth of the transformed layer after each experimental condition. The analysis was performed using an x-ray diffractometer (Bruker AXS, D8 Advance, Karlsruhe, Germany). Spectra were collected into the 2θ, with a range of 25-35º, at a step interval of 1 s and step size of 0.03º. The amount of m-phase (Xm) was calculated using the equation 1 [21]:

$$X_M = \frac{(-111)_M + (+111)_M}{(-111)_M + (111)_M + (111)_T} \qquad \text{Eq. (1)}$$

where: $(-111)_M$ and $(111)_M$ represent the intensity of the monoclinic peaks (2θ=28° and 2θ=31.2°, respectively) and $(111)_T$ indicates the intensity of the respective tetragonal peak (2θ=30°). The volumetric fraction (Fm) of the m-phase was calculated following the equation 2 [22]:

$$F_m = \frac{1.311 \cdot X_M}{1+0.311 \cdot X_M} \quad \text{Eq. (2)}$$

The depth of the transformed layer was calculated using the equation 3:

$$PZT = \left(\frac{sen\theta}{2\mu}\right)\left[ln\left(\frac{1}{1-FM}\right)\right] \quad \text{Eq. (3)}$$

where θ=15° (the angle of reflection), μ=0.0642 is the absorption coefficient, and FM is the amount of m-phase obtained using Eqs. 1 and 2.

Statistical analysis

Shapiro-Wilk test was used to test the normal distribution of the data. As the data (MPa) were normally distributed, bond strength data were submitted to 3-way analysis of variance (ANOVA) and Tukey's post-hoc tests (Statistix 8.0 for Windows, Analytical Software Inc, Tallahassee, FL, USA). *P* <0.05 was considered to be statistically significant in all tests.

Results

Overall, bond strength results were significantly affected by the particle size factor (p=0.015) but not the time (p=0.4034) and distance (p=0.1786) (3-way ANOVA). Interaction terms were significant (p<0.05).

Air-abrasion with 110 μm silica particles (10.96) presented significantly higher bond strength (p=0.015) compared to 30 μm (8.96) regardless of deposition conditions (Table 2). Air-abrasion with 110 μm particles (12.3) promoted significantly higher bond strength than that of 30 μm (6.4) when applied for 10 s from a distance of 10 mm (p<0.05, Tukey`s).

For 110 μm silica particles, increase in time from 10 to 20 s and distance from 10 to 20 mm did not increase the bond strength compared to 30 μm silica particles.

Phase transformation after air-abrasion ranged from 30.3 to 35.9% for 30 μm particles and 23.8 to 43.7% for 110 μm particles (Table 2). Prolonged duration of air-abrasion for 20 s presented the highest phase transformation at both 10 (43.3%) and 20 mm (43.7%) distances.

SEM analysis showed that the topography of Y-TZP surface altered after all air-abrasion protocols (Figs. 1a-f). In the groups, air abraded with 110 μm particles, larger surface area was evident.

Failure types were predominantly adhesive (Table 2, Figs. 2a-c). One cohesive failure was experienced in 110 μm silica coated group after 20 s air-abrasion.

Discussion

The impact of silica coated alumina particles on the Y-TZP surface increases roughness [2,23,24] through which micromechanical retention of the resin cements [2,4,5,7,8,19], and surface wettability is improved [9]. However, efficacy of air-abrasion procedures is dependent on the deposition parameters [18]. Based on the results obtained in this study, since particle type showed a significant effect on the results, the null hypothesis is rejected.

SEM analysis clearly revealed rough Y-TZP surfaces after air-abrasion with 110 μm particles compared to 30 μm particles. Larger particle size wears out the ceramic surface, approximately to the square of the abrasive size [25,26]. This may explain the higher bond strength for 110 μm than with 30 μm, at 10 s of deposition from a distance of 10 mm. With these parameters, the particle factor had significant effect on the adhesion. According to the results of a recent study, particle size did not influence the adhesion when aluminum oxide or silica coated aluminum oxide particles [27]. Nonetheless, in that study, bonded specimens were not aged through long-term water storage.

In this study, prolonged duration of deposition with 30 μm particles from 10 to 20 s at 20 mm ($S_{30}T_{20}D_{20}$ group) increased the mean bond strength. On the other hand, for 110 μm particles, already 10 s of air-abrasion at 10 mm ($S_{110}T_{10}D_{10}$ group) produced similar results. Thus, it can be stated that smaller particle size requires longer deposition duration to compensate for the factor of particle size to achieve a rough Y-TZP surface.

One problem associated with air-abrasion protocols is the t–m phase transformation resulting in volumetric expansion of zirconia grains. This generates compressive stresses at the Y-TZP surface, which may increase the mechanical properties [28] but also induce surface cracks [2,29]. In a previous study, 12 and 15% monoclinic phase was found after air-abrasion irrespective of the particle size and pressure [2]. In this study, higher percentages of t-m phase transformation (23.8 to 43.7%) were noted. The differences between the studies could be attributed to the variation in Y-TZP grain size [29]. Excessive phase transformation can result in material degradation, damaging the inherent mechanical properties of Y-TZP [19]. The maximum critical amount of monoclinic phase was reported to be 25% [29,30]. Thus, the results of this study indicate that both particles used for air-abrasion may degrade the mechanical behavior of the Y-TZP ceramic in the long run with the exception of $S_{110}T_{10}D_{20}$ group. Yet, the results need to be verified after fatigue tests.

In this study, the bonded specimens were exposed to both the thermal cycling and long-term water storage similar to previous studies [9,31-34]. The type of test method has also consequences on the magnitude of bond strength of resin cement to Y-TZP [12]. Shear bond strength test was used for the evaluation of adhesion in this study. One limitation of this kind of test is the development of non-homogeneous stress distribution at the bonded interface, especially when the knife-edge shaped loading device is used. For this reason, a wire-loop jig was used [35,36]. Each test method has its inherent shortcomings but for ranking purposes, currently, macroshear test has been the most commonly used method [12,37,38].

Failure type analysis showed the prevalence of adhesive failures being more frequent indicating that there is still need for improving adhesion of resin cements to Y-TZP ceramic. Future studies should also confirm the adhesion results as a function of surface roughness parameters.

Conclusions

From this study, the following could be concluded:

1. Surface conditioning of Y-TZP ceramic with air-abrasion prior to resin cement adhesion increased the bond strength with the increase in particle size from 30 to 110 μm. Except for air-abrasion with 30 μm silica particles, prolonged duration of air-abrasion up to 20 s and nozzle distance of up to 20 mm seem to have less impact on improved adhesion.
2. Both 30 and 110 μm silica coating created t-m phase transformation but 20 s of deposition in combination with 110 μm particle size resulted in the highest amount of monoclinical phase.

Clinical Relevance

For the adhesive cementation of minimal invasive Y-TZP dental reconstructions, chairside silica coating with 30 μm particles should be performed for 20 s form a distance of 10 to 20 s but for laboratory silica coating with 110 μm particles, 10 s of air-abrasion is sufficient.

Conflict of interest

The authors did not have any commercial interest in any of the materials used in this study.

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Captions to tables and figures:

Tables:

Table 1. Allocation of experimental groups according to the design of this study considering parameters of particle type, deposition duration, distance of the nozzle to the surface and abbreviation of the groups accordingly; S: Particle type; T: Time; D: Distance.

Table 2. Mean bond strength data (standard deviation) (MPa) for the tested groups, m-phase transformation (%) and transformation depth (μm). Different upper-case letters indicate statistically significant difference (Tukey`s, $p<0.05$).

Table 3. Frequencies of failure modes in percentage. Adhesive: Failure at the ceramic-cement interface with no remnants of the resin cement on the substrate; Cohesive: Cohesive failure in the resin cement.

Figures:

Figs. 1a-f Representative SEM photomicrographs of non-conditioned (a-c); air abraded Y-TZP surfaces with 30 μm particle size CoJet (g-i) and 110 μm Rocatec sílica coated alumina particles (left to right: x500, x1000, x2000). Note the increased roughness with the increase in size of the particles.

Figs. 2a-c . Representative micrographs of the failure modes from the tested samples. Image (A), (B) and (C) shows the adhesive failure mode at the ceramic-cement interface. The indicator (☛) represents the Y-TZP surface free of resin cement, while the pointer (☞) indicates the resin cement surface.

Figs. 3a-b Representative X-ray diffraction spectra for phase transformation analysis of the YTZP ceramic after **a)** air-abrasion with 30 μm CoJet sand, **b)** 110 μm Rocatec sand. Note the monoclic phase: lM(111) and lM(111); tetragonal phase: lT(111).